Elementary Electrochemistry
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Estimation of Total Chloride Ion Concentration in Triple Mixture using Primary Standard AgNO3

So, in the previous experiment I have shown you how one can do the estimation of chloride and HCL and ammonium chloride in a mixture of ammonium chloride HCL and potassium chloride using NaOH.

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So, now this is the second part of that experiment where we are going to estimate the concentration of total chloride in this mixture which we call as a triple mixture, mixture of

three different chlorides. So, we will estimate the concentration of chloride in this triple mixture using the primary standard that we have prepared is the standard silver nitrate solution. So, as you all know that when we add silver nitrate to a solution of chloride a curdy white precipate forms. So, you will see the same phenomena happening here.

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So, at the moment what we have taken in the beaker here is just some amount of distill water and you can see the conductance of that distill water is shown here it is about 8 micro-siemens, 8.72 micro-siemens.

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So, now I am going to pipette out this triple mixture and add it to this water for the estimation. As soon as I start adding this solution you will see that the conductance of the solution is changing, it will be increasing. So, it will take a while for the instrument to stabilize and equilibrate to the correct conductance of the solution which is solution of triple mixture.

So, before starting the experiment we should mix well and homogenize the solution so that it gives the correct reading for the conductance at the beginning. So, with zero volume of silver nitrate we need to take the reading of the conductance and we see the conductance is coming around 853.5 micro-siemens. So, now you can see that I have a slightly different burette, but it is again a 10 ml burette graduated.

You can see it is 10 ml graduated burette and I have filled this burette with the standard silver nitrate solution. So, from this I will start adding silver nitrate drop wise to my analyte solution in the beaker and you will see a curdy white precipitated forms when we add first time and further addition will continue increasing in that curdy white precipitated, but then you will not be able to see the difference so much because after addition we will have to shake it and that precipitated will be homogenized as a suspended particle in the solution.

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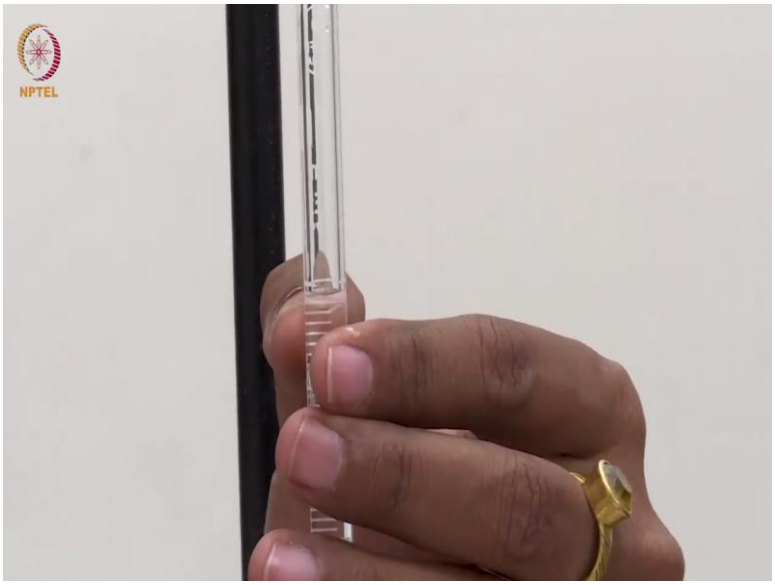

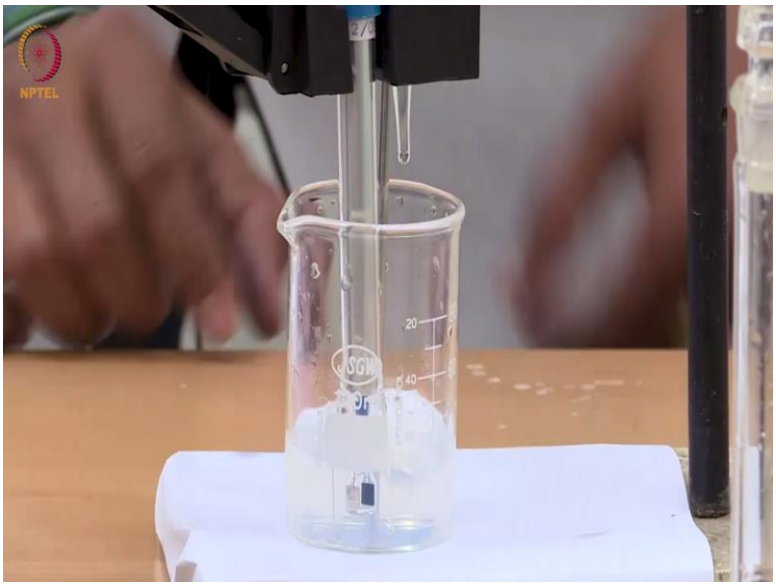

So, now I am going to add this silver nitrate to this solution. So, I will be adding very small amounts like 0.04 ml each time. You can see that with one drop of silver nitrate the solution has become curdy white the faint precipitated has formed. It is no longer a clear solution and that is because of the formation of silver chloride as precipitated. So, with 0.04 ml of silver nitrate you see the reading is not changing.

The conductivity value of this solution is not changing here at all. Why the conductivity is not changing because the reaction what is happening here is consumption of chloride ion by silver nitrate. So, chloride ion is removed as a silver chloride precipitated and is replaced by nitrate ion which has very similar conductivity compared to chloride and hence we will not see any significant change in conductance till we reach close to the endpoint.

Beyond the endpoint when we add we will see that the conductance is increasing significantly because of more and more silver chloride is added. So, in this particular titration we can use a larger volume gap between the two consecutive readings because from the theory we know there will not be any change in the beginning and there will be a change only when we have reached the equivalence point.

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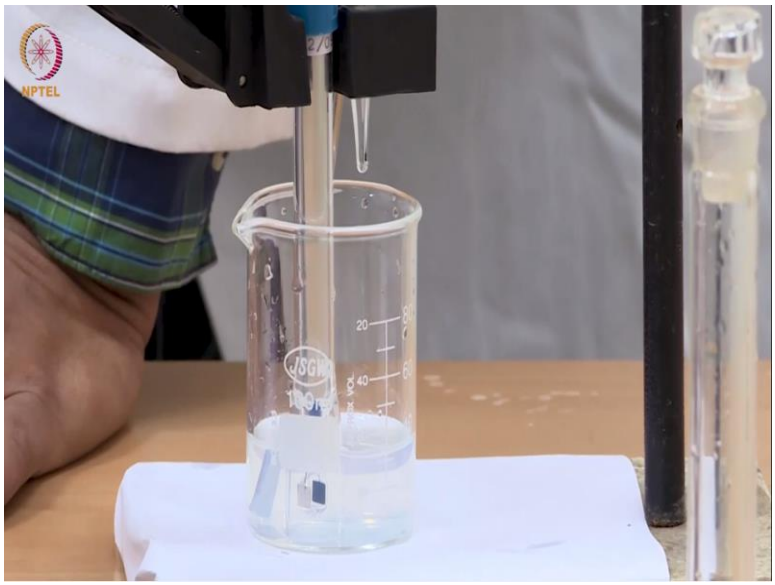

So, now I have reached a 0.1 ml and I should mix well so that the solution is homogenized. We will see that little more precipitated has formed and the solution is becoming more non transparent with time, but the conductance value it is not changing at all, it is still remains 852.2. This indicates that our experiment is going in the right direction.

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So, now I am going to add up to 0.25 ml. So, with 0.25 ml you can see further silver chloride formation which makes the solution more turbid and now we see a slight reduction in conductance 842.9 which is okay I think.

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So, we will now keep on adding 0.25 ml each time and take the reading. So, now we have reached 0.5 ml and the reading is slightly reduced, but then again it will equilibrate and give you approximately the same value as you know the conductance of chloride and nitrate is about the same. It is not exactly same and that is why we are seeing a slight gradual decrease. So, it is 842.6. We will add another portion of point, this burette is different.

So, I will add a 0.25 ml again. So with more amount of silver nitrate added you see the solution is becoming more and more turbid, white precipitate is forming and we should remember that this white precipitate can get deposited on the electrode. So, we have to do this titration very quickly and as soon as the titration is over we had to clean the electrode.

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So, we have reached a point when we have added 0.75 ml of silver nitrate and we are getting the conductance of 834, 835.2 maybe we just wait for a while 835.2.

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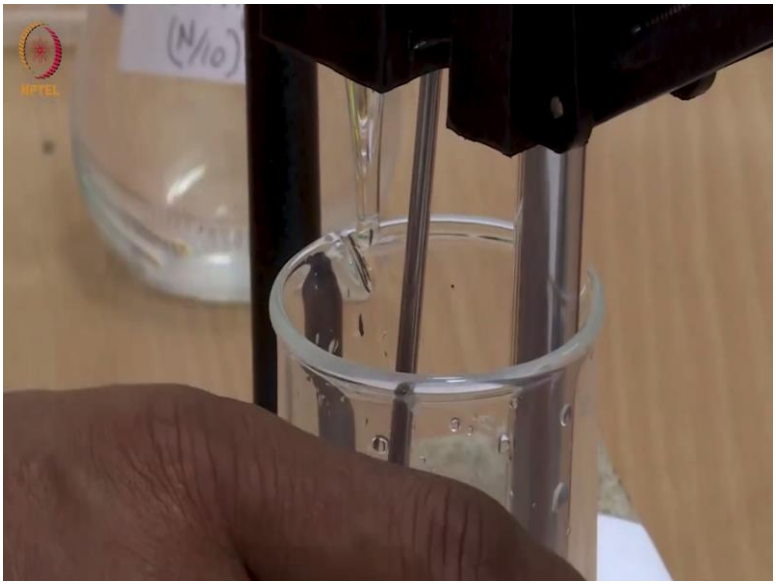

We will add one more portion of 0.25 ml. So, we compare to the previous experiments of both potentiometry and conductometry here our step size is more because we are in a region where we are expecting a nearly flat straight line where the conductance is not decreasing significantly. You see that we have already added 1 ml of silver nitrate and the conductance is now showing here in the conductivity cell it is 829.

So, from 0 to 1 ml which is now very close to the equivalence point the decrease in conductance has taken place from 853 to 829. So, with 1 ml addition the change is very small. If you go back to the previous video you will see that on addition from 0 to 0.5 to 1 there were significant changes in the conductance values and that is why we have to follow the steps of 0.02 or 0.04 ml.

But in this case since the variation is not so much we can have a larger gap between the two consecutive readings. So, now I am guessing that we are very close to the neutralization or equivalence point and after another one or two readings we will start seeing that the conductance value increases and then we will take a call at what point one should stop the addition.

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So, now I have added one more portion of 0.25 ml and going to start. You should be seeing the reading which is now showing stabilizing and then again giving you the value we need to homogenize for a while and wait. Now, you see as I was expecting there is an increase in conductance, the conductance has increased to 884.6 it is still increasing maybe it will stabilize at around 885 or slightly more than that which essentially indicates the precipitation titration has ended. The equivalence point has been reached crossed. So, we will take this reading as 885 where we see the first increase in conductance.

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Now, we will keep on adding 0.25 ml as before and take the drop in solution, mix well, shake well. So, now what is happening is all the chloride ions are consumed, whatever we are adding we are adding AgNO3 and the conductance increasing is corresponding to the amount of silver plus and nitrate ions that are added on top of whatever ions were present in this solution from the very beginning.

So, we will see that it has increased to 978, maybe 979, so 1.5 ml it is 978.6. So, it is in the increasing trend. We will need 2 or 3 more readings so that we should be able to plot a nice straight line after the equivalence. Before the equivalence we have so many points so with those we will be able to plot a straight line. After the equivalence also we will need at least 5, 6 points to draw a best fit straight line.

We have reached 1.75 ml and you see now it has jumped from micro-siemens to the region of milli-siemens. So, we should write it again in terms of micro-siemens. So, 1.073 milli-siemens means 1073 micro-siemens.

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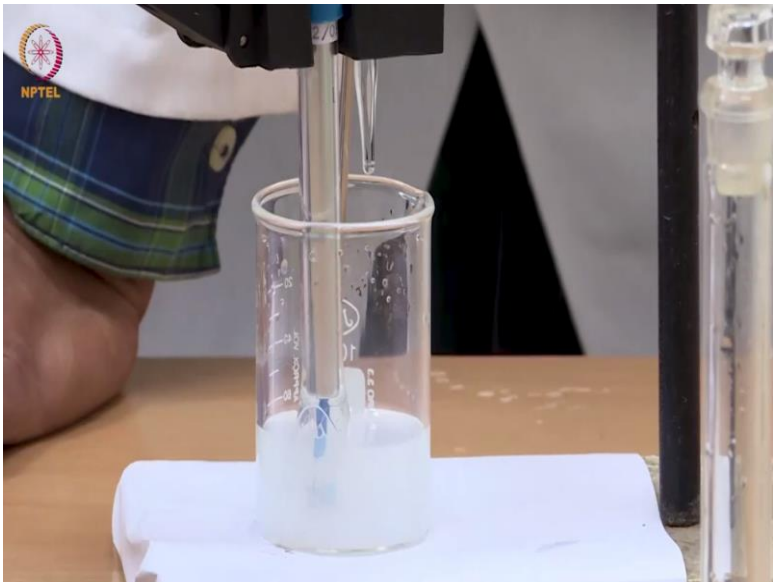

We will take one more reading with 2 ml and we see that this is coming out to be 1155 milli-siemens, micro-siemens. So, now if you see beyond about 1 ml which is just after the equivalence point the conductance was increasing. After 1 ml it is 1.25 is 85 from 885 to 978 is about 90, 978 to about 1,073 is again about 80. So, this constant increase 80 to 90 milli-siemens, micro-siemens indicates that we have got 3 or 4 points which are after the equivalence point.

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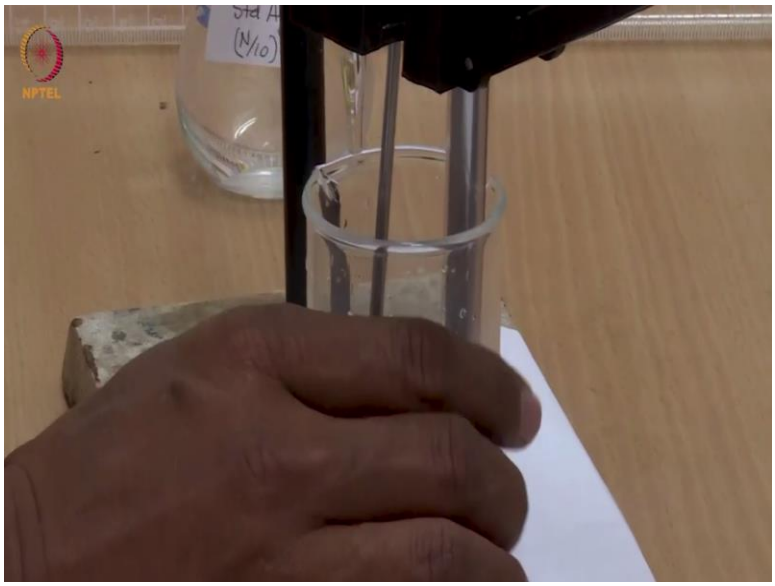

And we will just for the safety purpose we will take one more reading with another addition of 0.25, so we have 2.25, because at the end when we try to plot we should not be following short of the number of points. One or two points may go out of the straight line so we would need about 4 to 5 points to draw a best fit straight line. So, with 2.25 it will stabilize and give us the reading in a while.

You see now the response has become slow and that is simply because of a lot of silver chloride that is forming and it is probably getting deposited of the electrode and that is why it is bit slow in giving us the conductance value. So, it is 1233. So, we will stop at this point.

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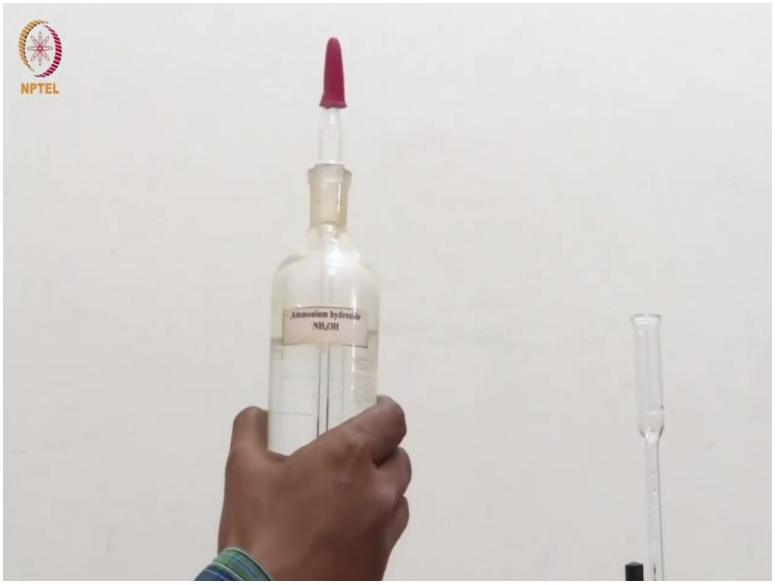
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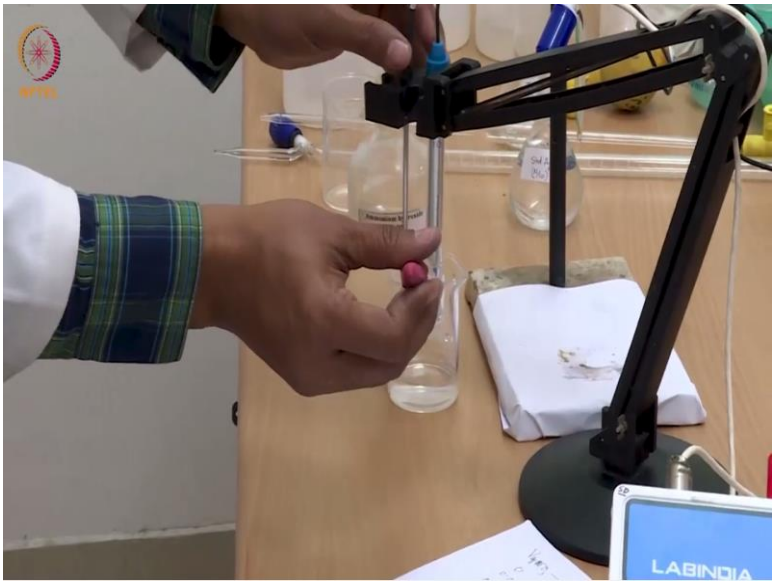
Ammonium hydroxide
NH4OH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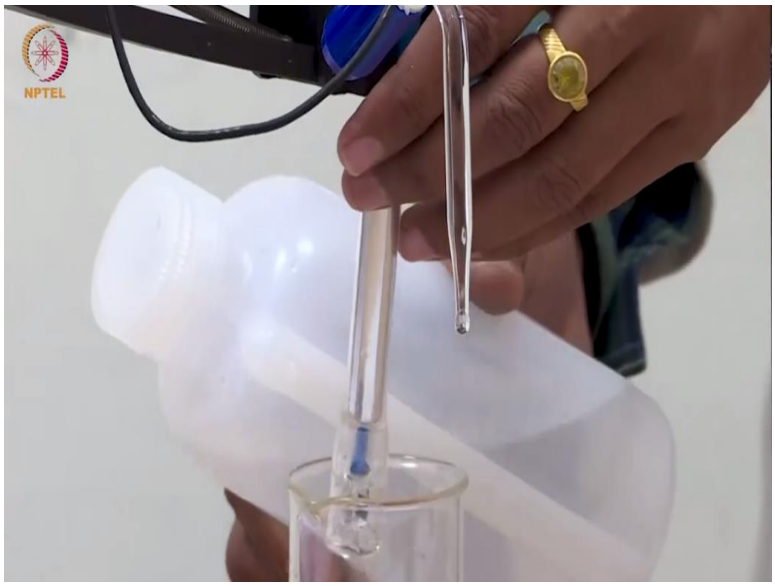
LABINDIA


NPTEL

Very quickly we should remove the burette, we should take it away and carefully lift up the electrode, remove the solution and throw it away. We take a separate beaker first to quickly wash the electrode with distill water and you know that distill water is not enough it cannot dissolve silver chloride which may have got deposited in this electrode. So, we should use a dilute solution of ammonium hydroxide which dissolves the precipitate of silver chloride.

And it will remove it from the electrode. We should be careful while using ammonium hydroxide, we should not smell it as you know it has a pungent smell, we are using a very dilute solution of ammonium hydroxide to dissolve any precipitate that maybe there sticking to the wall of the electrode. We should clean the electrode surface as well 2 to 3 times with this ammonia solution.

Now, we should wash this electrode thoroughly with water and remove all ammonium chloride or ammonium hydroxide that maybe here. This is a very, very important part of this experiment that you should wash the electrode carefully, so that you do not leave any silver chloride as deposited particles on the electrode which will then cause problem in the next set of experiments which we do or somebody else would do.

Whether the electrode is now clean or not one can see it by dipping it in distilled water. If the electrode is nice and clean we should get the starting conductance value which we already noted that it was about 8 micro-siemens. So, what we see here is that the conductance is much higher, it is about 200, 300 micro-siemens that essentially means the electrode is not fully clean yet.

So, we need to wash the electrode several times and discard that water, use more water to clean and keep an eye on the conductance value that it is showing. See now it has reduced to about 16 to 20 micro-siemens and still stabilizing which means it is getting cleaned, maybe one more round of cleaning with water jet it will be completely clean and we will see a reduction in conductance to about 8 to 9 micro-siemens.

You see now we have reached about 10 which is reasonable number maybe 1 or 2 more rounds of cleaning will reduce the conductance to 8 from where we actually started. So, I think this is a point where we need to leave because it is reading about 8 to 9, 10 it is alright. One can take this electrode to the lab and dip it in little more of ammonium hydroxide solution and remove.

But in my opinion if it is about 8 to 9 then I think it is fine. So, we have completed the experiment of triple mixture, we have done two titrations once with sodium hydroxide and once with silver nitrate and we have got the set of readings. So, in the next class I will discuss about this readings how to plot and what would be the result of these experiments. So, we will continue from here in the next theory class. Thank you.